NATO UNCLASSIFIED

NORTH ATLANTIC TREATY ORGANIZATION

ORGANISATION DU TRAITE DE L'ATLANTIQUE NORD

MILITARY AGENCY FOR STANDARDIZATION (MAS)
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4021

MAS/92-MMS/4021
18 April 1983

To : See distribution below

Subject : STANAG 4021 MMS (EDITION 3) - SPECIFICATION FOR CE (TETRYL) FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

References : a. MAS(ARMY)(62)640 dated 25 September 1962 (Edition 2)
b. AC/310-D/9 dated 1 April 1982

Enclosure : STANAG 4021(Edition 3)

1. The enclosed NATO Standardization Agreement which has been ratified by nations as reflected in page iii is promulgated herewith.

2. The references listed above are to be destroyed in accordance with local document destruction procedures.

3. AAP-4 should be amended to reflect the latest status of the STANAG.

ACTION BY NATIONAL STAFFS

4. National staffs are requested to examine page iii of the STANAG and, if they have not already done so, to advise the Defence Support Division of the International Staff, through their national delegation as appropriate of their intention regarding its ratification and implementation.

J.J.A. DOUCET
Major-General, CAAR
Chairman, MAS

DISTRIBUTION

Action: All members of the Army Board, MAS, except UK (for onward transmission to national authorities); UK - Director Standardization (STAN 2)

Information: SECGENNATO (DS Div); SACEUR; SACLANT; CINCHAN; CINCNORTH; CINCENT; CINCSOUTH; NAMSA

STANAG 4021
(Edition 3)

NORTH ATLANTIC TREATY ORGANIZATION
(NATO)

MILITARY AGENCY FOR STANDARDIZATION
(MAS)

STANDARDIZATION AGREEMENT

SUBJECT: SPECIFICATION FOR CE (TETRYL) FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

Promulgated on 18 April 1983

J.J.A. DOUCET
Major-General, CAAR
Chairman, MAS

RECORD OF AMENDMENTS

No.	Reference/date of amendment	Date entered	Signature

EXPLANATORY NOTES

AGREEMENT

1. This NATO Standardization Agreement (STANAG) is promulgated by the Chairman MAS under the authority vested in him by the NATO Military Committee.

2. No departure may be made from the agreement without consultation with the tasking authority. Nations may propose changes at any time to the tasking authority where they will be processed in the same manner as the original agreement.

3. Ratifying nations have agreed that national orders, manuals and instructions implementing this STANAG will include a reference to the STANAG number for purposes of identification.

DEFINITIONS

4. Ratification is "The declaration by which a nation formally accepts the content of this Standardization Agreement".

5. Implementation is "The fulfilment by a nation of its obligations under this Standardization Agreement".

6. Reservation is "The stated qualification by a nation which describes that part of this Standardization Agreement which it cannot implement or can implement only with limitations".

RATIFICATION, IMPLEMENTATION AND RESERVATIONS

7. Page iii gives the details of ratification and implementation of this agreement. If no details are shown it signifies that the nation has not yet notified the tasking authority of its intentions. Page iv (and subsequent) gives details of reservations and proprietary rights that have been stated.

RATIFICATION AND IMPLEMENTATION DETAILS
STADE DE RATIFICATION ET DE MISE EN APPLICATION

NATION	NATIONAL RATIFICATION REFERENCE DE LA RATIFICATION NATIONALE	NATIONAL IMPLEMENTING DOCUMENT NATIONAL DE MISE EN APPLICATION	IMPLEMENTATION/MISE EN APPLICATION					
			FORECAST DATE PREVUE			ACTUAL DATE DATE REELLE		
			NAVY MER	ARMY TERRE	AIR	NAVY MER	ARMY TERRE	AIR
BE	GS 5540 of/du 7.10.82						4.83	
CA	2441-4021(DAME 2) of/du 18.9.87	D-01-060-001/ AX-000					9.87	
DA	M.212.5-31P/MM2- of/du 26.8.83					1.84		1.84
FR								
GE	BMVg-Fü S IV 1 Az 03-51-60 of/du 9.7.82					8.83	8.83	8.83
GR	5707/21/4873 of/du 7.10.82					4.83		
IT	USG Prot.N. 423/4-70948- STANAG of/du 29.5.92						9.83	1.93
LU	GS 5540 of/du 7.10.82						4.83	
NL	M87/0198/0436 of/du 12.2.87					4.84	4.84	4.84
NO								
PO								
SP	NORMAT/0015/4021/03-00 of/du 14.2.91	STANAG				1.92	1.92	1.92
TU	4770(MAS)352 of/du 4.2.83							
UK	UK Del.Ltr of/du 30.8.82	DEF STAN 07-26/1				4.83	4.83	4.83
US								

Amendment 1

Agreed English/French texts

STANAG 4021
(Edition 3)

NAVY/ARMY/AIR

NATO STANDARDIZATION AGREEMENT
(STANAG)

SPECIFICATION FOR CE (TETRYL) FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

Annex : A. Melting Point Bath

Related Documents : None

AIM

1. The aim of the Agreement is to establish a common minimum specification for deliveries of CE(Tetryl) from one NATO nation to another.

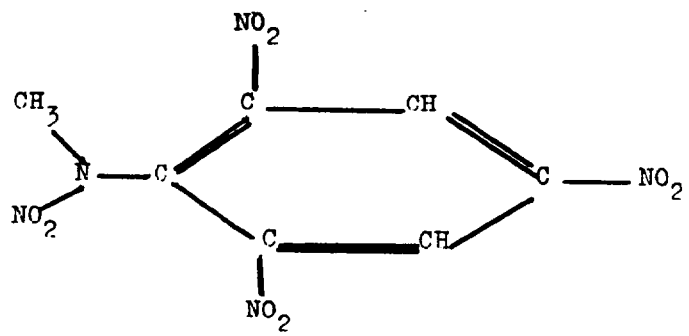
AGREEMENT

2. Participating nations agree that all CE manufactured by NATO nations to be delivered to another NATO nation, except when ordered for particular uses, must comply with the following minimum requirements.

PART I - PHYSICAL, CHEMICAL AND SAFETY CHARACTERISTICS

COMPOSITION

3. The CE (Tetryl) shall consist essentially of trinitrophenylmethylnitramine according to the formula:



APPEARANCE AND GRANULATION

4. The material shall be in the form of small yellow crystals (crystallised CE) or of aggregates of such crystals (granulated CE) as specified in the contract which may stipulate also a sieving test and a bulk density.

CHEMICAL DATA

5.	a.	Melting point	: min 128.8°C : max 129.5°C
	b.	Volatile matter	: max 0.10%
	c.	Insoluble in benzene	: max 0.07%
	d.	Inorganic matter	: max 0.03%
	e.	Acidity (as nitric acid)	: max 0.005%

SAFETY CHARACTERISTICS

6. Gritty particles: Following extraction with acetone and sieving as described in paragraph 15 not more than 5 gritty particles per 50 g sample shall be retained on a sieve of aperture 0.0098 inch (0.25 mm) and no gritty particle on a sieve of aperture 0.0165 inch (0.42 mm).

PART II - TESTING METHODS

GENERAL

7. Normal laboratory procedure applies to the determination of the characteristics of the materiel. The following descriptions are limited to the definition of particulars to be complied with when making tests.

PHYSICAL TESTS

8. Bulk density (when applicable): Determined on a 50 g sample placed in a 100 ml stoppered glass cylinder allowed to drop 50 times from a height of 2 1/2 inches (6.35 cm) on hard leather. The surface of the sample shall be levelled off by the minimum of side tapping in order to permit the reading of the volume.

CHEMICAL TESTS

9. Melting points. The melting point of CE (Tetryl) may be determined by either the Bloc Maquenne method or the capillary tube method. The method of test used shall be quoted on the test certificate.

10. a. Bloc Maquenne Method

(1) Apparatus:

(a) To determine the melting point of CE (Tetryl), an electrically heated Bloc Maquenne will be used, fitted with a mercury thermometer accurate to within 0.1°C over the whole thermometric scale.

10. a. (1) (b) Before being used, the Bloc Maquenne will be examined to ensure:

i. That the heating plate is not oxidised.

ii. That the rate of heating of the area selected for the determination of the melting point is constant and that the temperature is uniform in that area.

(2) Products used:

(a) In addition to the CE (Tetryl), of which the melting point is to be ascertained, pure benzoic acid and urea must be available.

(b) All the products shall be dry and pass through a sieve of aperture 0.0040 inch (0.10 mm).

(3) Method:

(a) Heat the Bloc Maquenne rapidly to about 115°C, then adjust the heating to obtain a rise in temperature of 1°C every three minutes.

(b) As from 120°C and keeping strictly to the above mentioned rate of heating, place on the plate of the Bloc Maquenne a few crystals of benzoic acid (melting point provisionally stated to be 122.37°C).and renew them at least every thirty seconds until the melting point of the product is reached within 10 to 15 seconds. The moment this occurs, read temperature T_1 on thermometer. Continue in this manner with the Tetryl to be tested and subsequently with the urea (melting point provisionally stated to be 132.70°C). Temperature T_2 and T_3 respectively are thus obtained on the thermometer.

(c) Calculate the differences:

$T_1 - 122.37^{\circ}C = a$

$T_3 - 132.70^{\circ}C = b$

a and b should be less than 1°C and (a-b) less than or equal to 0.1°C. If the latter two conditions are not met the reason(s) must be found. It will be necessary to check the satisfactory operation of the Bloc Maquenne, to examine the thermometers.

(d) The melting point of the CE(Tetryl) is given by $T = T_2 - a$.

10. b. Capillary Tube method

(1) Apparatus:

(a) To determine the melting point of CE (Tetryl) the apparatus detailed below will be used.

i. Thermometer, short range, short stem thermometer, full immersion type, having a range of 125 - 135°C, graduated at 0.10°C intervals.

ii Melting point bath, shown in Figure A-1. It consists of a tall form 800 ml beaker of resistance glass (A) closed with a chloroprene bung (B) through the centre of which passes a glass tube (C). The tube is closed with a chloroprene bung (D) through the centre of which passes the shaft of a mechanical stirrer (E) which shall be such that the direction of the flow of the liquid is downward. Four holes (F) in the tube allow circulation of the heating liquid.

The bung (B) has a hole to accommodate the thermometer (G) and, at a distance of about 1 cm small holes are bored on either side to accommodate melting point tubes (H).

The bath is filled with silicone oil or other suitable liquid to such a depth that the liquid circulates freely when the temperature of the bath is within 10-15°C of the melting point.

iii. A suitable screen is necessary to protect the apparatus from draughts when in use.

iv. Melting point tubes (H). Capillary tubes 150 mm long and 0.5 to 1.0 mm internal diameter.

v. A second thermometer is necessary to indicate ambient temperature.

(2) Product used. A portion of the sample shall be ground with caution to a fine powder and dried in a boiling water oven for 2 hours.

10. b. (3) Method:

(a) Transfer to the dry capillary tube sufficient of the ground and dried sample to fill it to a depth of 1 cm. Immerse the thermometer in the bath so that the bottom of the bulb is about 40 mm above the bottom of the beaker. Support a second thermometer, with its bulb as near as possible to the exposed stem of the main thermometer.

(b) Run the mechanical stirrer so that the liquid flows downwards in the tube (C), and regulate the speed, about 600 revs/min., so that the surface of the liquid between (C) and (A) remains level.

(c) Raise the temperature of the bath at a rate of $10^{o}C$ per minute by means of a bunsen burner, taking care only to heat the base of the beaker, and protecting the flame and bath from draughts.

(d) When the temperature of the bath is about $10^{o}C$ below the expected melting point of the sample, insert the capillary tube containing sample in one of the small holes in bung (B), using a small paper "flag" for support if necessary, so that the bottom of the tube is level with the bottom of the thermometer bulb.

(e) Reduce the heating rate so that the temperature rises at $1^{o}C$ per minute. The material may slump and change colour as the melting point is approached. This appearance should not be confused with the first appearance of liquid.

(f) Note the temperature T_1 at which liquid first appears and the temperature T_2 at which the material has just fully melted. Apply any necessary corrections for thermometer bore error.

(g) Calculate and report the melting point as follows:

$$\text{Melting point } {}^{o}C = \frac{(T_1 + C_1) + (T_2 + C_2)}{2} + C_3$$

(h) Where C_1 and C_2 are the corrections for thermometer bore errors at readings T_1 and T_2 respectively, and $C_3 = 0.00016N\ (T-t)$ where N = number of degrees divisions of exposed mercury column T=indicated temp in deg C and t=average ambient temperature, indicated by a second thermometer whose bulb is near the mid-point of the exposed mercury column of thermometer (G).

11. Volatile matter. To be determined from the loss of weight of 20 g of the material placed in a flat bottomed dish - 2 to 3 inches in diameter - placed for 6 hours in a boiling water oven. The dish shall be provided with a closely fitting lid when cooling and weighing.

12. Insoluble in benzene. To be determined on 10 g of the material dissolved in 200 ml of dry benzene at boiling temperature. Filter on a tared Gooch or sintered glass crucible. Wash the crucible thoroughly with dry benzene. Dry it in a boiling water oven for 1 hour, cool and weigh.

13. Inorganic matter. To be determined by incinerating the residue insoluble in benzene.

14. Acidity:

a. Weigh exactly 10 $\pm$ 0.01 g of dry tetryl and place it in a 500 ml extraction flask.

b. Add 50 ml of acetone measured in a 100 ml graduated test tube.

c. Shake by hand from time to time until the explosive is completely dissolved.

d. Pour in slowly (20 to 30 seconds) 200 ml of distilled water measured in a 250 ml test tube and wait until the explosive settles (about 10 minutes).

e. Add 8 to 10 drops of methyl red/methylene blue indicator (0.1 g of methyl red and 0.05 g of methylene blue in 100 ml of 95 % ethyl alochol) and titrate at once, without filtering using 0.05 N sodium hydroxide. For this purpose use a 5 ml semi-micro-burette graduated in 1/50 ml, 1 ml corresponding to a length of 70 to 80 mm.

f. Add the 0.05 N sodium hydroxide solution drop by drop to the flask, shaking until the indicator end-point is reached.

g. Note the volume V_1 of solution used.

h. Carry out a blank test simultaneously under identical conditions to those of the actual determination. For this purpose, place in a 500 ml or larger extraction flask 50 ml of acetone, 200 ml of distilled water and 8 to 10 drops of methyl red/methylene blue indicator. Titrate with 0.05 N sodium hydroxide solution. Note the volume V_2 of solution used.

14. i. The acidity of the tetryl expressed as percentage of nitric acid is given by:

$$0.0315\ (V_1 - V_2)\ f$$

where f is the correction factor for the normality of the sodium hydroxide solution.

SAFETY TESTS

15. Gritty Particles: Transfer a weighed portion of approximately 50 g to a sieve of aperture 0.0098 inch (0.25 mm). Place the sieve in a Soxhlet apparatus or other suitable extractor. Add sufficient acetone and extract on a steam bath until all the CE is dissolved. Remove the sieve, count and examine the remaining particles. Brush the particles on to a sieve of aperture 0.0165 inch (0.42 mm), count and examine any that are retained.

IMPLEMENTATION OF THE AGREEMENT

16. This STANAG is considered implemented when a nation has issued the necessary orders/instructions putting the contents of this agreement into effect.

MELTING POINT BATH - *BAIN DU POINT DE FUSION*

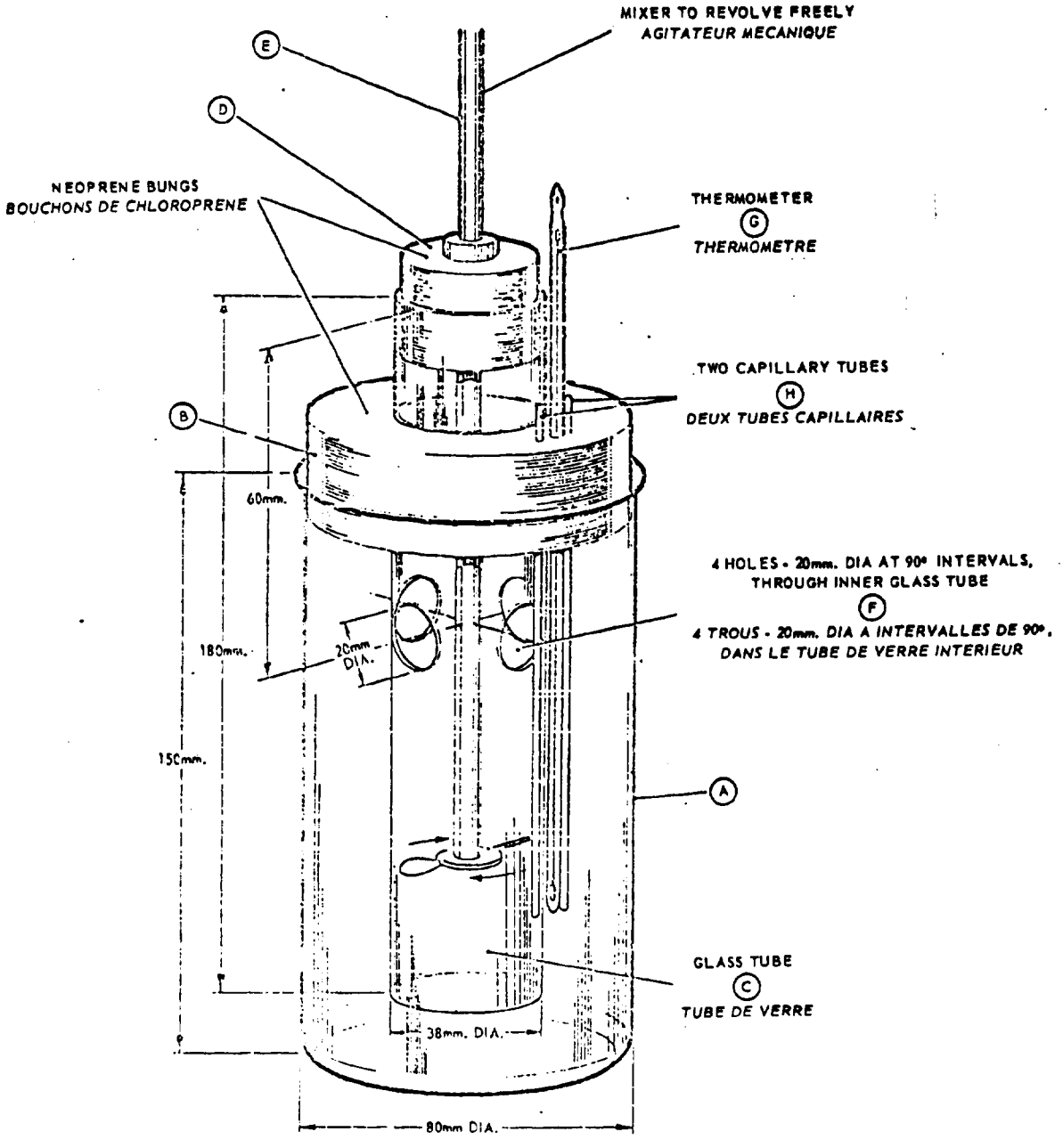


FIGURE A-1